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EVALUATION OF COMMERCIALLY SUPPLIED SILVER COATED TEFLON FOR SPACECRAFT TEMPERATURE CONTROL USAGE

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JAMES B. HEANEY

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GODDARD SPACE FLIGHT CENTER
GREENBELT, MARYLAND

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ABSTRACT

This report describes a series of tests performed to evaluate the acceptability of a commercially supplied silver backed teflon thermal control coating relative to teflon previously coated at GSFC. Optical measurements made on numerous samples indicate that the commercial material possesses an average solar absorptance of 0.085, an emittance of 0.76 and an average α/ϵ equal to 0.112, all of which are equivalent to the GSFC coated teflon. The emittance of the protective inconel backing was found to be 0.037. The coating is shown to have good adhesion at the Ag-teflon interface and exposure to uv irradiation uncovered no coating irreguiarities. Temperature cycling over the range -135°C to +200°C produced crazing in the evaporated Ag layer as expected but no delamination was observed. The suitability of Mystik #7366 and 3M #467 adhesives as bonding agents for the metallized polymer is demonstrated. Various problems associated with production reproducibility and selection of a proper bonding process are discussed.

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EVALUATION OF COMMERCIALLY SUPPLIED SILVER COATED TEFLON FOR SPACECRAFT TEMPERATURE CONTROL USAGE

INTRODUCTION

In the past several years metallized FEP teflon has emerged as a popular spacecraft temperature control material. Recent publications have demonstrated that clear sheets of FEP teflon type A, 125 μm (5 mils) thick and coated with a reflective layer of evaporated Ag plus a protective layer of inconel on the rear surface, possess solar absorptances (α) as low as 0.075 with emittances (ϵ, 300 K) of 0.76 and α/ϵ values as low as 0.10.[1,2] Qualification tests performed both in the laboratory and in space have shown that this coating is extremely resistant to damage from ultraviolet irradiation and is moderately stable in a charged particle environment.[3,4,5] In actual flight situations, thermal control surfaces coated with Ag-backed teflon (Ag/teflon) have operated within design limits over a period of years in orbit, although some problems associated with anomalous temperature increases have been observed.[6,7,8] The observed temperature increases are anomalous in that they exceed predictions derived from laboratory tests and they therefore serve to highlight the complexity of the connection between the total system—coating plus adhesive—and total environment—temperature, vacuum and irradiation.

By mid-1972, spacecraft thermal design engineers at the Goddard Space Flight Center (GSFC) had acquired substantial flight and laboratory practical experience with teflon, the major portion of which had been coated with Ag plus inconel in-house by Goddard's Engineering Applications Branch. Goddard coated Ag/teflon was used extensively on the Small Astronomy Satellite (SAS) series, portions of the Orbiting Astronomical Observatory (OAO-III), Interplanetary Monitoring Platform (IMP-I) and other spacecraft and in addition, was flown on two Thermal Control Coatings Monitors, one aboard the Orbiting Solar Observatory (OSO-H), the other on IMP-I, specifically intended to study Ag/teflon's actual degradation in space.

The growing demand for this coating resulted in a decision to seek an outside, commercial source of supply. This report describes an investigation undertaken to guarantee that Ag coated FEP teflon procured from a commercial supplier* was equal to or better than the GSFC coated material. The investigation was aimed at establishing confidence in a new source of supply while permitting its immediate usage on spacecraft without the delays of repetitious testing. For this

*G. T. Schjeldahl Company, Northfield, Minnesota 55057.

reason, qualification tests were designed to be comparative in nature, matching the performance of samples selected from the commercial material against control samples taken from teflon coated at GSFC. The intent was to allow all previous test data as well as design and handling experience gained with the latter coating to be directly applied to the commercial material.

Silver coated teflon procured from G. T. Schjeldahl Co. has been (or will soon be) used by GSFC personnel as a thermal control material on the Mariner-Venus-Mercury spacecraft magnetometer, booms, and plasma science experiment; Helios magnetometer; SAS-C; Radio Astronomy Explorer (RAE-B); OSO-I; International Ultraviolet Explorer (IUE); IMP-J and others. Hundreds of square meters of spacecraft exterior surfaces have been or will soon be covered with this commercially purchased material. Although the primary intent of the investigation was to guarantee general coating comparability, test parameters were matched with specific spacecraft needs whenever possible.

In the report that follows, each area of investigation is treated as a complete section in itself, containing the test objective, methods used and results obtained. Conclusions and comments are collected in a final summary.

The following topics will be discussed.

A. Sample Selection, Identification and Visual Examination.

B. Quick Metal Adherence Check (Scotch tape test).

C. Optical and Radiative Property Measurements.

D. Ultraviolet Irradiation Test.

E. Temperature Cycling Test.

F. Conclusions and Comments.

A. Sample Selection, Identification and Visual Examination

The purchased material consisted of clear sheets of DuPont's FEP type A teflon coated on one side with vacuum deposited Ag plus a protective layer of evaporated inconel #600 alloy. Ag/teflon was also obtained in the form of a tape with adhesive and release cover (3M #467) already applied. The teflon in all cases was 125 μm (~5 mils) thick. Silver coated teflon without adhesive was received in sheets 1.22 m wide by 3.05 m long (4' x 10'); tape dimensions were approximately 2.54 cm or 5.08 cm in width and up to 32.9 m (108') in length.

Samples were selected with the intent of establishing the uniformity of the material's properties over a large area, or along a roll, and the reproducibility of

these properties among sheets. Since each sheet or roll is the product of a separate evaporation process requiring the precise control of many variables, the potential for non-reproducibility is real.

The sample selection scheme used to achieve a degree of randomness with a relatively small number of samples is depicted in Figure 1 for the large sheets. Each piece cut out was approximately 15.2 cm by 15.2 cm (6 in. x 6 in.) square. Test samples were taken from the tape rolls at the beginning, 1/4 of the way in, near the middle and near the end and were each about 5 cm long.

Figure 1 also demonstrates the identification scheme. Each sample can be identified by its sheet number (I, II, III, etc.) and location on the sheet (IA, IIC, etc.). Letters A, B, C, D refer to the upper right corner, bottom right offset 15 cm from edge, middle, and left edge 2/3 of the way down from the top, respectively. Letter E refers to a randomly located badly scuffed area from sheet 1. Only two tape rolls were studied, identified as TI and TII, and sample location is spelled out when needed.

A sample identified as "control" was selected from a sheet of teflon that had been coated with evaporated Ag plus inconel by GSFC's Engineering Applications Branch. This sample was chosen as representative of the type of material that GSFC engineers have been using for the past several years in flight applications. All tests were designed to be comparative in nature, to establish a connection between the newer purchased coating and the type previously prepared at Goddard.

This selection operation produced 21 separate samples plus 1 "control" for subsequent examination. All identified samples served as a source of numerous smaller specimens, not further identified, that were subjected to the various tests.

Visual examination of the Ag/teflon was performed at the time of sample selection. Except for a narrow ($\sim$ 1 cm) strip around the perimeter of each rectangular sheet caused by gradients in evaporated film thickness, there were very few observable imperfections over the whole 3.72 m^2 (40 $ft.^2$) area. Occasional "scuff type" marks were seen, apparently caused by imperfections in the teflon itself, and sample IE was chosen to be representative of these, as mentioned above. Tape samples contained a large number ($>$ 50/cm) of parallel striations running the length of the roll. Since this trait was uniformly present in both rolls, it was accepted as a characteristic of the material in this form rather than an imperfection of the type.

The material was also examined for visible transparency and number of pinholes by holding the sheets in front of a bright light. (Microscopically small areas of teflon not coated with Ag or inconel are referred to as pinholes. There were

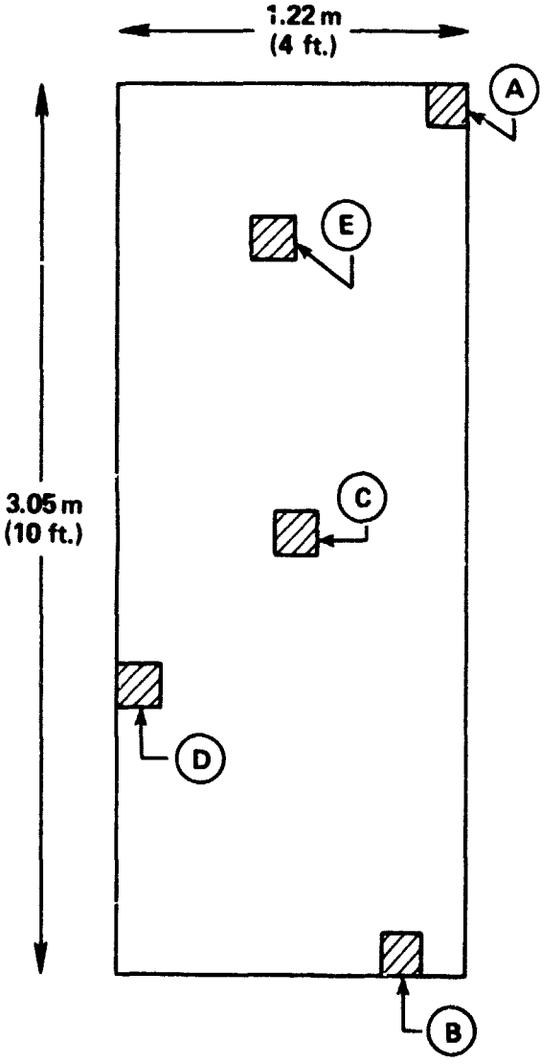


Figure 1. Location of Samples Selected
From 1.22 m by 3.05 m (4' x 10') Sheets

no holes in the teflon sheet.) Passing a fluorescent lamp behind the coated teflon in a darkened room is an effective way to enhance the visual appearance of pinholes. There was a noticeably larger number of pinholes in the commercial material than in the control sample. This fact has no significant impact on initial radiative properties such as solar transmittance or absorptance. However, numerous pinholes can be responsible for a more rapid aging, especially in a sulfur rich environment. This is demonstrated in Figure 2 where reflectance decrease is plotted as a function of wavelength after 6 hours exposure to an

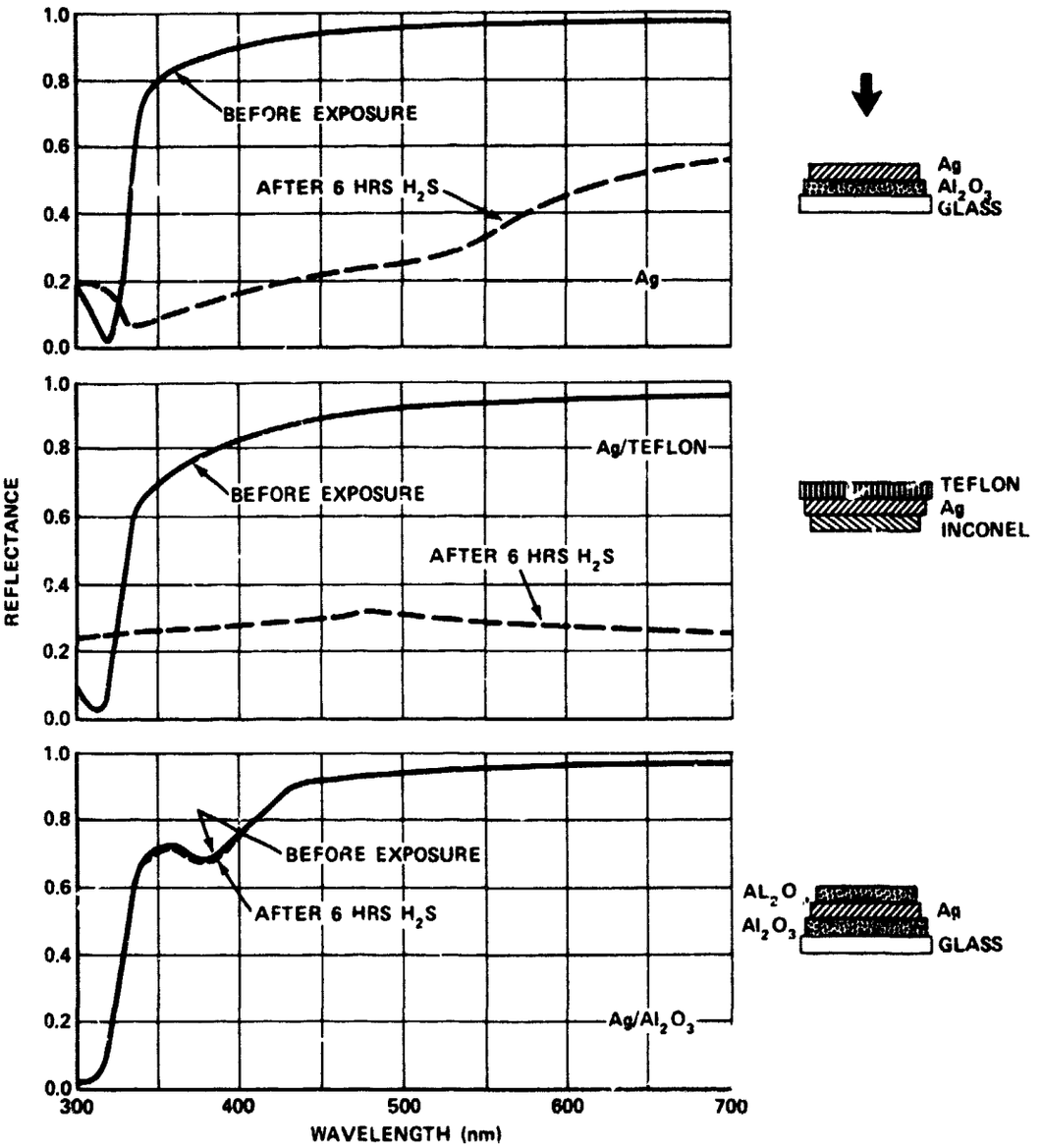


Figure 2. Reflectance of Ag/Teflon, Unprotected Ag and Ag Protected With Thin Evaporated Al_2O_3 Before and After H_2S Exposure

enriched H_2S atmosphere. The severity of the environment can be gauged from the performance of the other 2 Ag coated reflectors also plotted in Figure 2. The first coating consists of exposed Ag evaporated onto glass with an intermediate Al_2O_3 layer; the third is an identical surface overcoated with a thin protective layer of Al_2O_3. Although the degradation indicated in Figure 2 is the result of an accelerated aging process, it nevertheless demonstrates the susceptibility of Ag/teflon to long term aging in a hostile sulfur environment. In the case of coated teflon, pinholes act as avenues to allow sulfur to pass through the protective inconel layer and degrade the Ag. For this reason, adhesives containing sulfur compounds should be avoided when bonding Ag/teflon to a substrate.

The Ag/teflon tape samples, rendered opaque by the adhesive backing, were not subjected to this type of examination, but presumably they contain an equivalent number of pinholes.

When viewed from the back surface (inconel coated side), there were apparent differences in the reflectances of the inconel layers. This was attributed to variations in deposition conditions (thickness, pressure, rate) during the inconel evaporation. Again, this easily observable difference does not directly affect the material's performance capabilities, but it does indicate a variance in preparation conditions. The reflectance of the inconel backing will be discussed in the section on Optical and Radiative Property Measurements.

B. Quick Metal Adherence Test

A standard technique for rapidly evaluating how well an evaporated coating adheres to its substrate consists in applying cellophane tape (3M Scotch) to the coated surface and then quickly pulling the tape away. If the coating is removed with the tape, then it is judged to be unsuitable for use. The test is obviously not definitive in a quantitative sense but it does constitute a rapid pass-fail criterion for weeding out unsuitable material. Passing such a test does not guarantee high quality but failure does indicate either an improperly performed evaporation or an incompatible combination of materials.

All 21 test samples passed the "Scotch tape test" but the control sample did not. A separate investigation determined that there was a correlation between poor adhesion and thickness of the Ag coating applied to teflon.[9] This statement oversimplifies the problem somewhat, since factors such as glow discharge prior to evaporation, rate of deposition, etc. must also be considered. However, the Ag film on the control piece of Ag/teflon was judged to be thicker than desired. This did not diminish its value as a reference standard, a fact that will be evident from the results of subsequent tests.

The Ag/teflon tape samples, TI and TII, were not subjected to this delamination check.

C. Optical and Radiative Property Measurements

Silver coated teflon 125 μm (5 mils) thick is generally considered to have a solar absorptance (α) of approximately 0.08 and a hemispherical emittance (ϵ) of about 0.76 at 300 K. Both α and ϵ vary directly with the thickness of the teflon film. A more comprehensive treatment of this topic has been given in earlier publications.[1,2] It was the intent of this investigation to determine α and ϵ for the commercially purchased Ag/teflon and to compare the values with those obtained from the control sample. These measurements would largely determine the uniformity and reproducibility of the material's radiative properties.

Solar absorptance values were obtained from spectral reflectance measurements performed over the wavelength region from 0.29 μm to 8.0 μm. The region from 0.29 μm to 2.4 μm was covered by using an integrating sphere reflectometer; a heated cavity (hohlraum) reflectometer was used to extend the measured limits of the solar wavelength region to 8.0 μm. Because the α of Ag/teflon is quite low ($\alpha \simeq 0.08$), even small measurement errors become significant from a percentage standpoint. For this reason, the methods used to measure solar reflectance and to calculate α are explained in more detail in an attached Appendix. The results of these measurements are presented in Table 1 for the indicated samples. The presence of the last sample in Table 1, Alzak, will be explained in a later section.

Near-normal room temperature emittance, ϵ_n (300 K), was determined using a Gier-Dunkle DB-100 portable infrared reflectometer.[10] This device measures total emittance directly and a separate integration of spectral data is not required. Hemispherical emittance of Ag/teflon can be obtained from near-normal measurements using Jakob's ϵ/ϵ_n conversion factor[11]. The ϵ_n data in Table 1 indicate good agreement among samples. If an ϵ_n value of 0.805 $\pm$0.010 is chosen as typical, then a value of ϵ = 0.76 is obtained from ϵ/ϵ_n = 0.945. This is in good agreement with calorimetric measurements performed on 125 μm (5 mils) thick Ag/teflon which yield an ϵ = 0.76 at 300 K.

Since the magnitude of ϵ is essentially determined only by the infrared absorption character of teflon, it is largely insensitive to variations in the quality of the reflective Ag layer. α, on the other hand, is dominated by the reflectance of the metal backing because teflon is highly transparent in the solar region. Table 1 indicates that α does vary and values as high as 0.097 were measured for the samples studies here. The data of Table 1 are intended to be comparative and for this reason it is important to mention that the α values contain a precision of $\pm$0.003. Accuracy, as indicated in the table, is $\pm$0.010 and is

Table 1

Optical Measurements and uv Degradation Data

Sample	α①	α_F②	$\Delta\alpha$	ϵ_N③	ϵ	α/ϵ
Control	0.085	0.088	+0.003	0.812	0.767	0.111
IA	0.085			0.804	0.760	0.112
IB	0.082			0.805	0.761	0.108
IC	0.084	0.085	+0.001	0.800	0.756	0.111
ID	0.080			0.799	0.757	0.106
IE	0.082	0.083	+0.001	0.802	0.759	0.108
IIA	0.097	0.097	0.000	0.809	0.765	0.127
IIB	0.087	0.085	-0.002	0.806	0.762	0.114
IIC	0.084			0.802	0.759	0.111
IID	0.080			0.803	0.759	0.105
IIIA	0.084	0.086	-0.002	0.807	0.763	0.110
IIIB	0.084			0.806	0.762	0.110
IIIC	0.083			0.807	0.763	0.109
IIID	0.091	0.090	-0.001	0.803	0.759	0.120
IVA	0.082			0.795	0.755	0.109
IVB	0.094	0.086	-0.008	0.801	0.757	0.124
IVC	0.088	0.086	-0.002	0.806	0.762	0.115
IVD	0.079			0.806	0.762	0.104
TI-(1/4 way in)	0.087			0.810	0.765	0.114
TI-End	0.087	0.093	+0.006	0.814	0.769	0.113
TII-Beginning	0.081	0.083	+0.002	0.807	0.763	0.106
TII-Middle	0.090			0.801	0.757	0.119
Alzak	0.158	0.178	+0.020			

① - Precision: ±0.003; Accuracy: ±0.010.

② - Measured after 200 equivalent uv sun hours.

③ - Precision: ±0.005; Accuracy: ±0.010.

discussed further in the appendix. Variations in α can be attributed as much to defects in the teflon film as to the quality of the Ag reflective layer, although it is interesting to note that the α of sample IE, a scuffed piece of Ag/teflon, is relatively low. In the worst case, i.e., sample set IV, non-uniformity was ±10%. Applying a standard deviation check to the α data of Table 1, excluding the control sample and Alzak, reveals that a ±6% variance can be expected. The ϵ data produced a value of 0.112 ±5%. This was judged to be acceptable and all samples were found to be satisfactory with respect to their α and ϵ values.

It was evident from an earlier visual examination that transmittance varied from sample to sample. The transmittance referred to here is that due to variations in the optical density of the Ag and inconel layers and not that due to pinholes. For light in the blue region of the visible spectrum, pinhole transmittance per unit area is several orders of magnitude less than that coming through the combined metal layers. The calculated transmittances of Ag and inconel as a function of thickness are presented in Figure 3 for 2 wavelengths. Transmittance at 400 nm is of course visibly evident. The curves of Figure 3 were calculated from the optical constants of Ag given in Reference 12 and from the optical constants of inconel obtained from Reference 13. Figure 3 is useful in placing upper limits on the unknown thicknesses of Ag and inconel applied to the Ag/teflon samples being tested. Ag obviously has a high transmittance at λ = 326 nm, even for films 1000 Å thick. The calculated transmittance of inconel is uniform over the selected wavelength range and is much less than that of Ag. It should be emphasized that Figure 3 is an idealization that may not be met in actual practice. This is demonstrated in Figure 4 where the spectral transmittances of selected Ag/teflon samples are given over the wavelength range from 280 nm to 400 nm. The measured data of Figure 4, supported by the calculated curves of Figure 3, clearly indicate that the optical density of the inconel and Ag layers varies from sheet to sheet. This means that the deposition conditions vary, although there is no evidence that they stray beyond acceptable limits.

It should also be noted that less than 0.10% of incident solar radiation is transmitted by the sample with the highest transmittance, IIA.

As mentioned above, visual examination had revealed variations in the reflectance of the inconel protective layer. The inconel on the control sample prepared at GSFC was noticeably brighter than any of the commercial source material. This is shown in Figure 5 where the reflectance of the inconel side of selected samples is plotted over the wavelength region from 0.30μm to 2.4μm. The specimen labeled S/N 6 was an especially dark piece supplied by G. T. Schjeldahl Co. and introduced for the purpose of comparison. This specimen was not from the group of samples undergoing evaluation but was inserted here because it is the darkest piece encountered to date and therefore represents the limit of our experience. The uv transmittance of S/N 6 can be found in Figure 4. The calculated reflectance

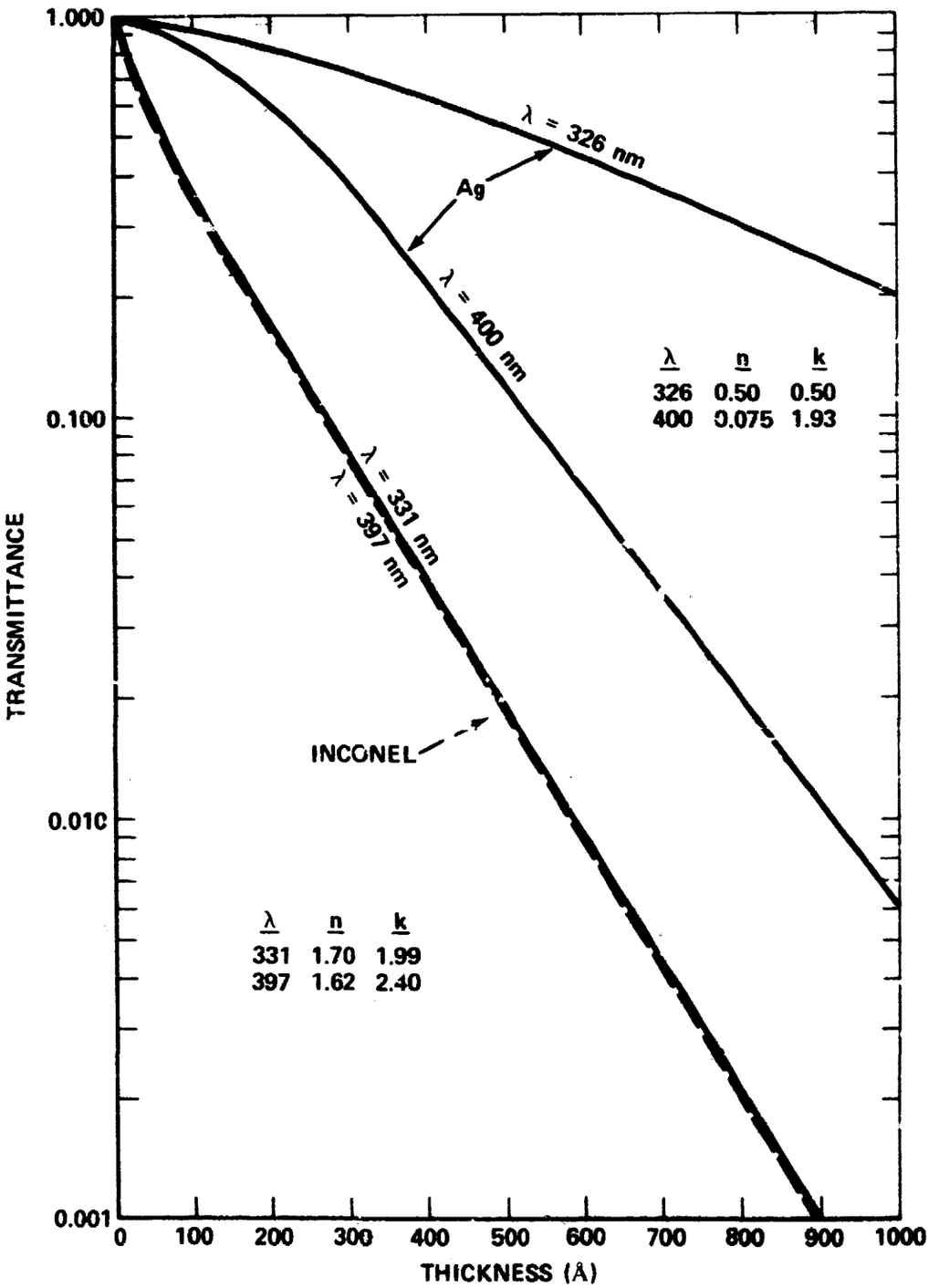


Figure 3. Calculated Transmittance of Ag as a Function of Thickness at λ = 326 nm and λ = 400 nm; Transmittance of Inconel #600 Alloy as a Function of Thickness at λ = 331 nm and λ = 397 nm

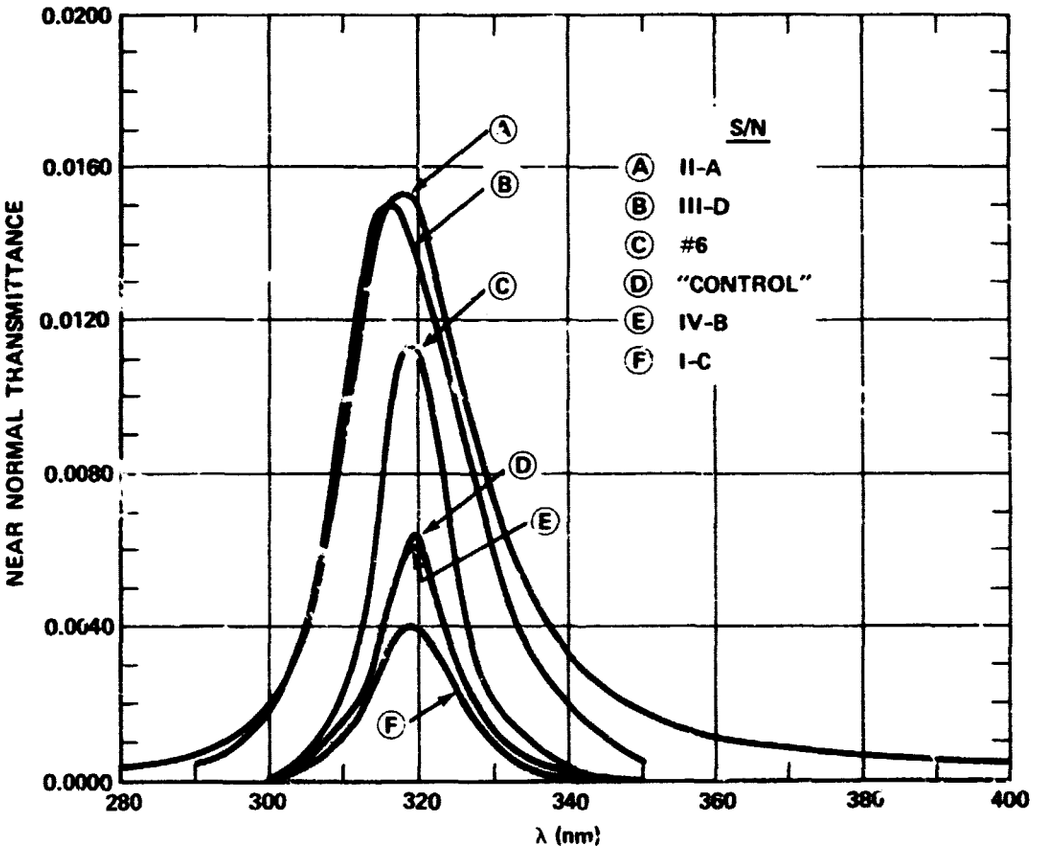


Figure 4. Transmittance of 125 μm (5 mil) Teflon/Ag/Inconel #600 Alloy Over the Wavelength Region From 270 nm to 400 nm for Selected Samples

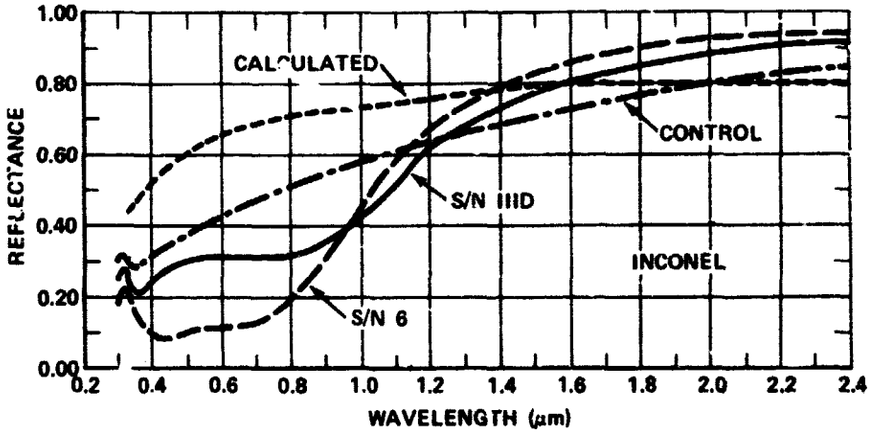


Figure 5. Reflectance of the Inconel Side of Selected Ag/Teflon Samples Over the Wavelength Range From 0.30 μm to 2.4 μm

curve of Figure 5 is based on optical constant measurements obtained from Reference 13 which relate to a film of inconel #600 alloy 625 Å thick. These referenced data indicate an increase in reflectance with thickness that is especially apparent at wavelengths greater than 1.0μm. Connecting these observations with the measured data of Figure 5 leads to the inference that the inconel backing on the measured samples is thicker than 625 Å. The low visible reflectance in the case of the test samples is most likely due to evaporation conditions—pressure, rate, etc.—and not caused by any post-evaporation degradation. Test specimens measured before and after an 11 month aging in an ambient laboratory environment, including exposure to fluorescent room lighting, exhibited less than 1% change in total reflectance. Some aging was evident, however, from a visually observed increased surface scattering apparently due to small scale irregularities forming on the surface and perhaps associated with pinholes as discussed above. The inconel film deserves separate attention because the protective benefit that the Ag layer derives from the inconel is directly translated into the long term stability of α and, to a lesser extent, ϵ.

Transmittance measurements have already indicated that deposition conditions probably vary during the evaporation of the Ag and inconel layers. The α data of Table 1, obtained by measuring the reflectance of the Ag backing through the clear teflon film, supports this interpretation of the results. Finally, Figure 5 reveals that the reflectance of the outer inconel layer also varies. This point is examined further with the aid of Figure 6. In Figure 6, the calculated reflectance and transmittance of teflon coated with Ag plus inconel is plotted as a function of inconel thickness for 3 specific thickness of Ag—500 Å, 750 Å and 1000 Å—at λ = 326 nm and with inconel as the surface of incidence. The teflon layer is assumed to be non-absorbing, which is a fair assumption that helps keep the results of Figure 6 consistent with those of Figure 3. Figure 4 has indicated that the Ag/teflon test samples have a peak transmittance of about 1% to 1.5% at $\lambda \simeq$ 320 nm. If this value is superimposed on Figure 6, and the Ag layer is assumed to be 500 Å thick, then the thickness of the inconel layer must be $\leq$ 400 Å. This already disagrees with the reflectance data of Figure 5. If the Ag is assumed to be thinner, the disagreement grows. It is apparent that the idealized calculated results of Figures 3 and 6 are not matched in reality by the Ag/teflon test samples. This is not an unusual occurence and can most probably be attributed to the less than ideal quality of the Ag and inconel layers on the test samples. Since defects induced by flawed evaporation are more evident at shorter wavelengths, this could explain the low visible reflectances of Figure 5, even for thick inconel films. Figure 6 also reveals that the reflectance of very thin inconel films ($t <$ 250 Å) on Ag is dependent on the Ag thickness at λ = 326 nm. It is unlikely that the inconel film would ever be this thin.

The near normal emittance of the inconel surface was also measured for selected samples and the results are given in Table 2. They show that ϵ_n is quite low

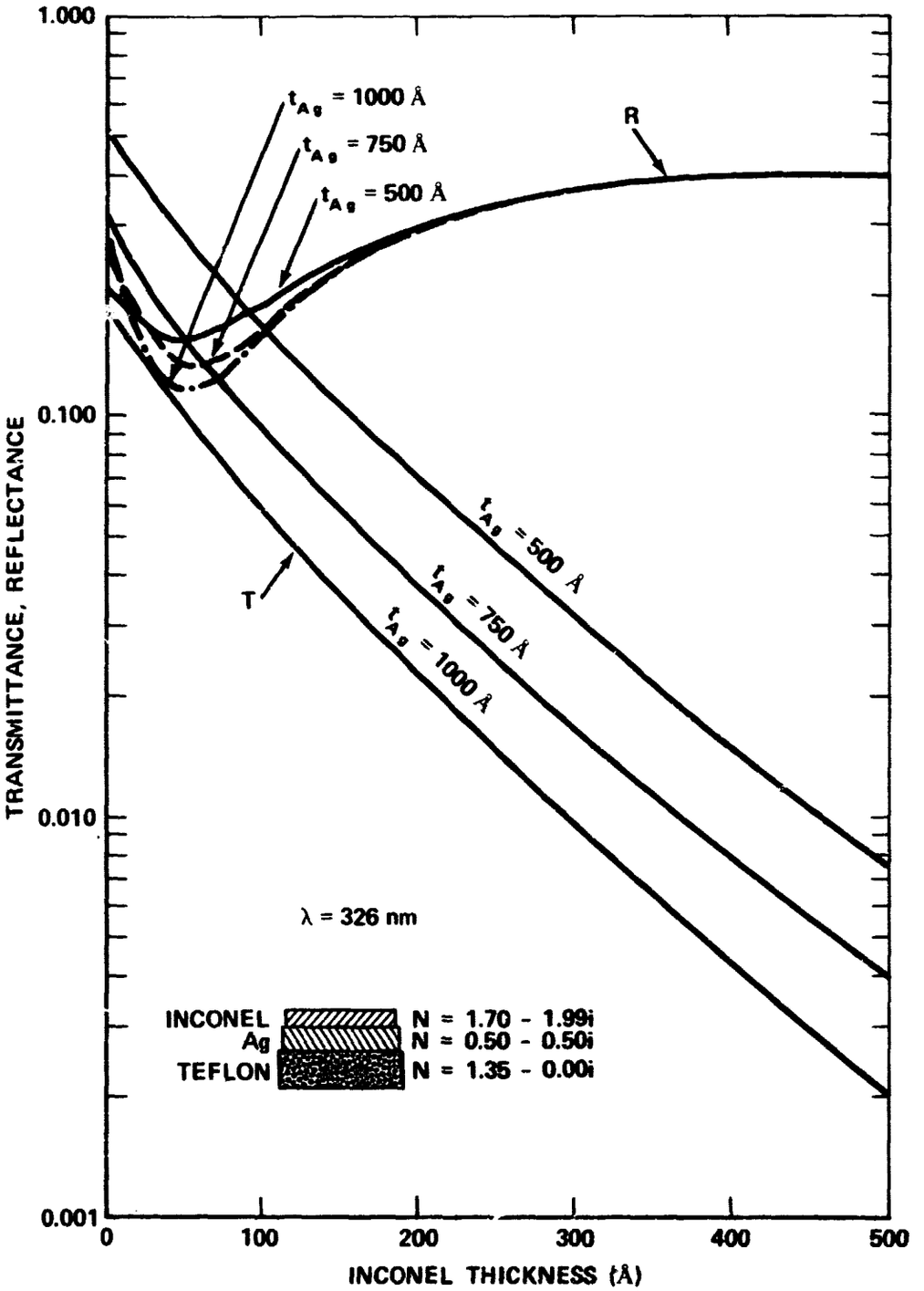


Figure 6. Calculated Reflectance and Transmittance of Teflon Coated with Ag plus Inconel as a Function of Inconel Thickness for 3 Specific Thicknesses of Ag—500 Å, 750 Å and 1000 Å—at λ = 326 nm

Table 2

Emittance of Inconel

Sample	ϵ_n ①	ϵ ②
Control	0.030	0.038
IC	0.029	0.037
IIA	0.027	0.035
IIID	0.027	0.035
IVB	0.029	0.037
#6	0.033	0.042
		$\bar{\epsilon}$ = 0.037

① - Precision: ±0.003, Accuracy: ±0.01

② - ϵ obtained using Jakob's value ϵ/ϵ_n = 1.28.

and doesn't vary despite the fact that the visible reflectances of the corresponding inconel surfaces noticeably different. ϵ was determined from ϵ_n using a conversion factor of $\epsilon/\epsilon_n = 1.28$ obtained from Jakob[11]. At $\epsilon = 0.037$, the back surface emittance of Ag/teflon is already low and it should not be necessary to reduce it further. The use of evaporated Al instead of inconel directly over the Ag film to achieve a reduction in ϵ should be avoided. Ag and Al in contact will *diffuse spontaneously, even at room temperature,* and lead to an eventual degradation in α.[14]

D. Ultraviolet Irradiation Test

Recent publications have established the fact that FEP type A teflon films are extremely resistant to damage from ultraviolet irradiation.[1,2,5,15] For this reason, if degradation were to be observed in Ag/teflon coatings exposed to ultraviolet irradiation, then the quality of the evaporation process during which the Ag reflective layer was deposited would be suspect. The main intent of subjecting the commerically purchased Ag/teflon to an ultraviolet exposure was to establish the relative merit of this material by comparing its rate of degradation to that of the control sample. A secondary purpose was to perform a simulated solar exposure test whose results would benefit future spacecraft on which the commercial Ag/teflon would be used.

Ten test specimens were chosen from the commercial source material—IC, IE, IIA, IIB, IIIA, IIID, IVB, IVC, TI and TII—as indicated in Table 1. The choice

of specimens was intended to serve the needs of uniformity, consistency and randomness within the constraints imposed by the test facility. During irradiation the specimens were located within separate compartments of a common vacuum chamber and were effectively isolated from one another. Each compartment had its own uv grade sapphire window possessing high transmittance to uv irradiation down to λ = 180 nm and lower. Vacuum in the main chamber was kept in the 10^{-6} torr range. All samples, including the control sample and Alzak, but excluding TI and TII, were bonded to water cooled substrates using double sided kapton tape (Mystik #7366). Specimens TI and TII were purchased with an adhesive already applied (3M #467) and were bonded directly to the cooled substrate. Sample temperatures were maintained within the range 20°C to 30°C throughout the test.

Ultraviolet irradiance was provided by a filtered xenon arc source (Spectrolab x-25). Two independent techniques were used to verify the equivalent uv solar content of the incident flux. First, total irradiance was monitored using a calibrated thermopile and a filtered Si photodiode was used to obtain the uv content of the beam. The Si cell plus filter combination was calibrated to yield solar equivalent flux in a broad band from 250 nm to 320 nm and in a narrow band around 380 nm. A second technique utilized the well defined degradation behavior of an Alzak sample to obtain a post-test estimate of the total ultraviolet dosage. The Alzak sample was snipped from a larger piece that has supplied test specimens to several flight experiments and carefully controlled laboratory tests. Its usage as an uv dosimeter in this manner is described in Reference 5.

The irradiation test was performed until a total exposure corresponding to 200 solar uv equivalent hours was reached. Solar absorptance data were obtained in air before and after exposure. It had previously been determined that neither teflon nor Alzak experienced rapid recovery upon a post-irradiation exposure to air. The results of the α measurements are presented in Table 1. The behavior of the specimens taken from the commercial material was equivalent to that of the control sample. A decrease in α (increase in ρ_λ) produced by an ultraviolet irradiation of relatively short duration such as this is not unusual for Ag/teflon and has been previously noted.[2,16] Specimen TI, a tape sample, showed the largest degradation but its $\Delta\alpha$ was small and well within acceptable limits for a test of this duration. The precision of the $\Delta\alpha$ data of Table 1 is derived from spectral reflectance changes that are 3 or 4 times larger at some wavelengths than the indicated value of $\Delta\alpha$ and is sufficient to allow this distinction to be made wih regard to sample TI.

The Alzak sample had a $\Delta\alpha \simeq 0.02$ which agrees with the radiometric estimate of a total exposure corresponding to 200 equivalent uv solar hours.

There is no easy explanation for the observed variance in $\Delta\alpha$ among samples. Measurement precision (see α data), minute traces of surface contamination and the undefined processes manifested in the initial decrease in α could be separately or collectively responsible for the observed difference. This point was not pursued as part of this investigation.

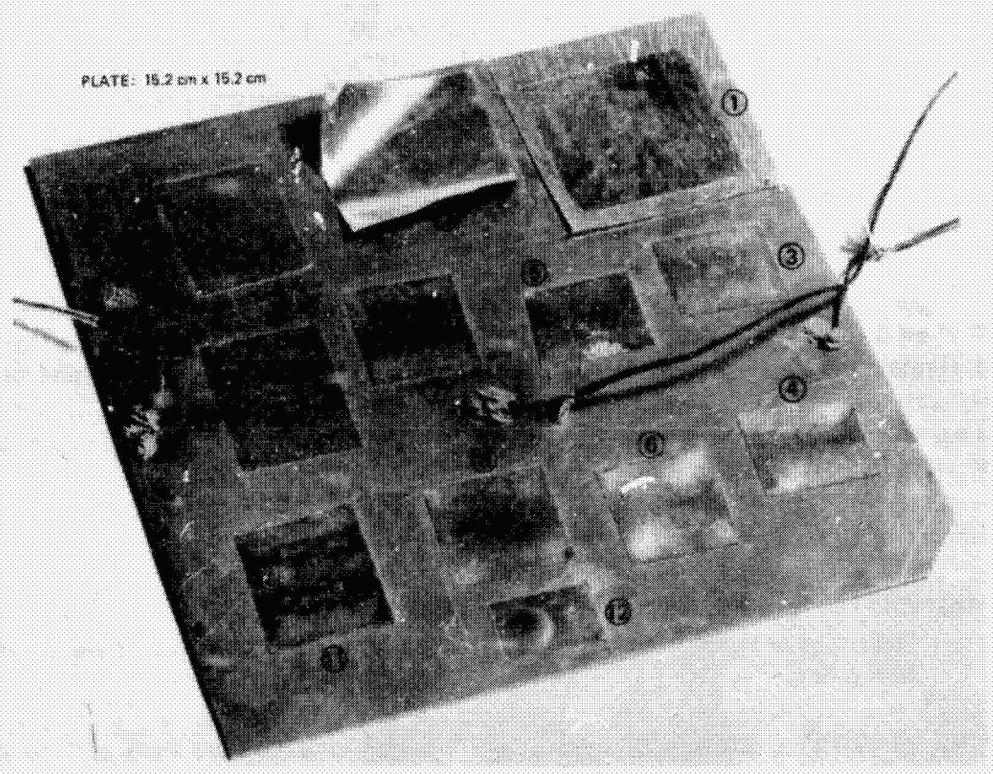
E. Temperature Cycling Test

In order to evaluate the Ag/teflon bond strength in a manner more realistic than the quick "scotch tape" adhesive check described above, the commercial Ag/teflon was subjected to a temperature cycling test. Although the thermal test was not primarily aimed at evaluating the performance of adhesives used to bond the Ag/teflon samples to the test plate, this information was generated as a by-product.

Four different adhesives were used—3M #467 which was supplied already applied to the Ag/teflon (samples TI and TII), Mystik #7366, Gelva 263 (thinned 1:1 by volume hexane: ethyl acetate) and RTV 6 - 1104 (diluted with toluene). Mystik #7366 has been the adhesive most frequently used to bond Ag/teflon to GSFC spacecraft and flight experiments; 3M #467 is a candidate substitute. The last 2 adhesives were chosen solely for experimental purposes.

All the test specimens were bonded to the same aluminum substrate with the various adhesives as shown in Figure 7. Each sample-adhesive combination is also identified in Figure 7. Two specimens were added here to examine applications problems. S/N 11 consists of a single piece of teflon placed down on 3 strips of #7366 adhesive separated by about 3 mm. This arrangement subjected the bonded and unbonded areas to unequal stresses resulting in a fracturing of the Ag film over the unbonded portion. Applying heat to this sample allowed the fracturing to be studied as a function of temperature. The Mystik #7366 tape used in this test was "outgassed" prior to bonding the various Ag/teflon specimens. Outgassing was accomplished by applying each piece of adhesive to the Al sheet with their release covers intact and then setting the sheet aside at room temperature in the laboratory for 2 weeks. At that point, the tape release backing was removed and the Ag/teflon specimens were applied. In order to demonstrate what happens if the Ag/teflon is bonded to #7366 tape that has not first been aged in this manner, S/N 12 was prepared by placing a piece of Ag/teflon down on #7366 tape taken directly from the roll. No high temperature cure or other preparatory procedure was used on any of the specimens.

Since the "scotch tape" test had revealed poor adhesion at the Ag/teflon interface in the case of the control sample, a newer GSFC supplied specimen with a thinner Ag layer was added to the temperature cycling test array. This is identified as S/N 8—GSFC(2) in Figure 7.



LOCATION			IDENTIFICATION
①	S/N IB	+	Gelva 263 adhesive thinned 1:1 (vol.) hexane : ethyl acetate.
②	S/N ID	+	RTV 6-1104 diluted with toluene.
③	S/N IB	+	Mystik #7366
④	S/N IIC	+	Mystik #7366
⑤	*CONTROL	+	Mystik #7366
⑥	S/N IIIB	+	Mystik #7366
⑦	S/N IVA	+	Mystik #7366
⑧	GSFC (2)	+	Mystik #7366
⑨	S/N TII	+	3M #467 (Adhesive applied to Ag/teflon by supplier).
⑩	S/N TI	+	3M #467 (Adhesive applied to Ag/teflon by supplier).
⑪	S/N IVD	+	Mystik #7366 applied in 3 separate strips.
⑫	CONTRCL	+	Mystik #7366 with no prior in-air aging or outgassing.

Figure 7. Photograph of Test Samples After Thermal Cycling Test, Including Identification and Description of Ag/teflon-Adhesive Combination

The Al substrate was mounted on a temperature controlled plate in a glass vacuum bell jar which was then pumped down to the 10^{-5} torr range. Temperature cycling was conducted in 4 stages—+50°C to -136°C, +100°C to -95°C, +150°C to -120°C and +200°C to -15°C—in order to approach potential problems while minimizing the chance of bypassing a critical point. Visual observation was maintained during thermal cycling through the bell jar and the chamber was opened to permit closer inspection after each cycle. The sample plate was horizontal, samples facing up during the test.

The 4 cycles are graphically displayed in Figure 8 where temperature is presented as a function of time. The progress of each cycle is plotted in the figure with flagged points indicating noteworthy events. These events are defined in summary form below. Before proceeding into this summary it should be noted that an unfortunate chamber failure caused by a ruptured seal in a liquid nitrogen line prematurely ended the test at the -15°C point in cycle 4.

Summary of events occuring during thermal cycling:

a. At -112°C in the first cycle, S/N 1 began to lift extensively at 1 corner. Delamination and cracking of the Ag was also evident. S/N 1 failed at this point.

b. At -20°C in the second cycle, S/N 2 began to lift up and curl at 1 corner. The Ag did not crack or delaminate but came away intact. S/N 2 failed at this point.

c. Between +108°C and +115°C in cycle 3, S/N 2 lifted extensively and curled along corners and edges. Approximately 50% of the total area had lifted. Warping occurred with temperature variations for the remainder of the test.

d. At +135°C in cycle 3, a corner lifted slightly on S/N 8. The lifting was confined to less than 5% of the total area.

e. At +70°C on the cool down slope of cycle 3, S/N 10 exhibited a slight lift along 1 edge, confined to less than 2% of the total area.

f. At -15°C in cycle 4, a liquid nitrogen connection separated inside the chamber causing a rapid compression that ended the test. This compression produced microscopic fracturing in the Ag backing preferentially located in certain well defined areas.

A post-test photograph of the specimens mounted on the Al sheet is given in Figure 7. Note that the corner piece containing S/N 1 had been sawed from the

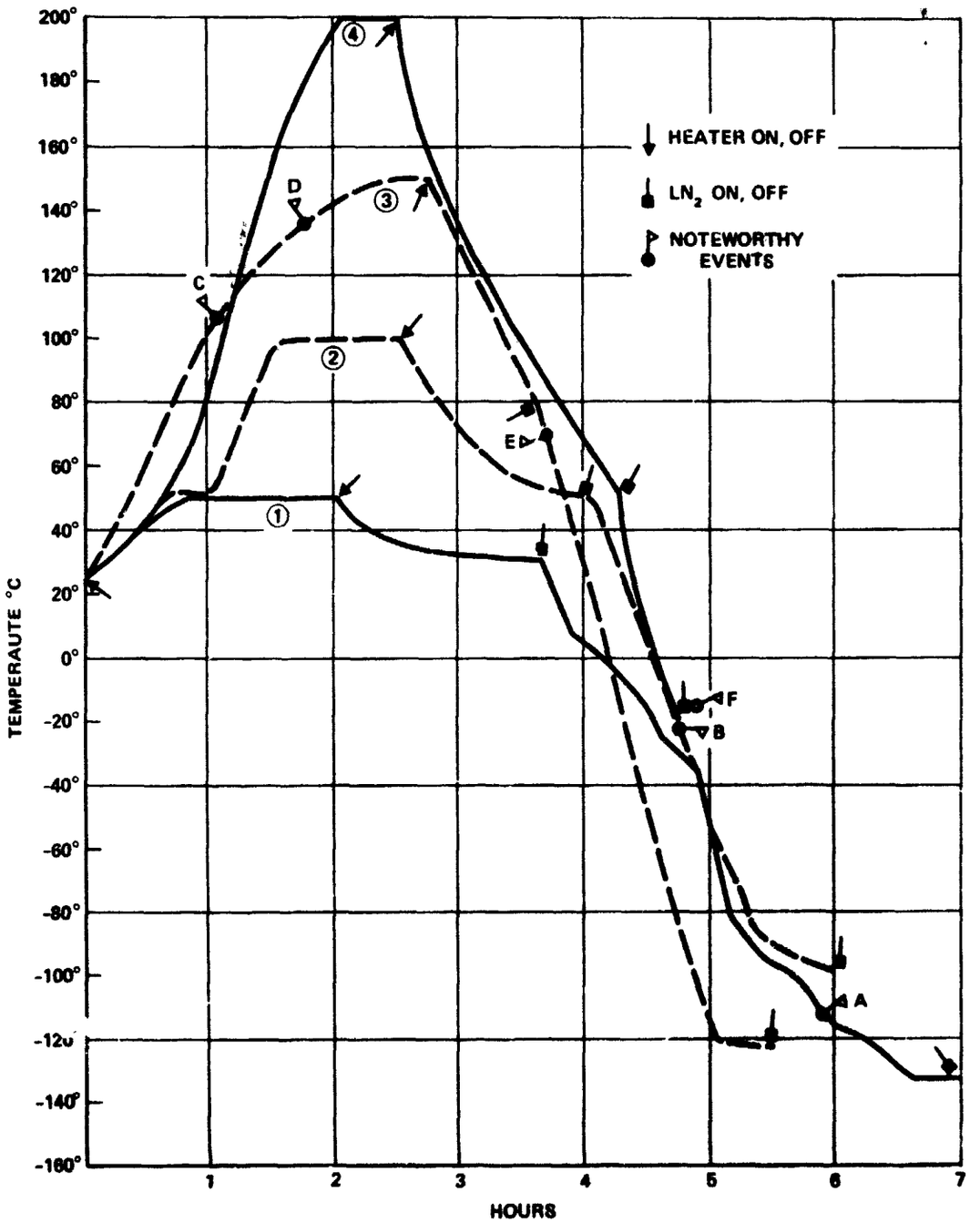


Figure 8. Variation of Temperature with Time for 4 Cycles

plate prior to being photographed. The Ag film of S/N 1 had separated from the teflon in some places and had lifted from the adhesive in others. The Ag was badly fractured. S/N 2 has obviously lifted. Specimens 3 through 10 remained in good contact with the plate. The fracturing of the Ag film on certain specimens that occurred during the unexpected chamber venting is highlighted by the contrast between bright and dark areas. All the test samples and even the test plate are quite specular which accounts for the contrast irregularities in the photograph.

A photograph of the crazed area of S/N 6 enlarged to 160 x is presented in Figure 9. This type of effect was common to all specimens but was much less pronounced on S/N's 9 and 10 (i.e. TI and TII, bonded with 3M #467). The crazing, identified by the "map" cracks in Figure 9, was caused by thermal cycling. This occurs when the Ag/teflon is heated above 125°C (approximately) and has been observed and discussed in earlier work.[17] It is very similar in appearance to the mechanical fracturing of Ag that occurs whenever Ag/teflon is bent sharply, stretched or pulled away from an adhesive. This effect can increase α by about 0.03 and is responsible for an increase in scattering and loss of specularity of the Ag/teflon coating.[17] In viewing S/N 11 in Figure 7, the 3 strips of underlying adhesive are easily recognized. The 2 intermediate clearer areas are unbounded and were extensively flexed by thermal cycling. The Ag/teflon part that was unbonded exhibited crazing at an earlier stage in the test than any other sample. Slight crazing was in fact evident at the end of cycle 2.

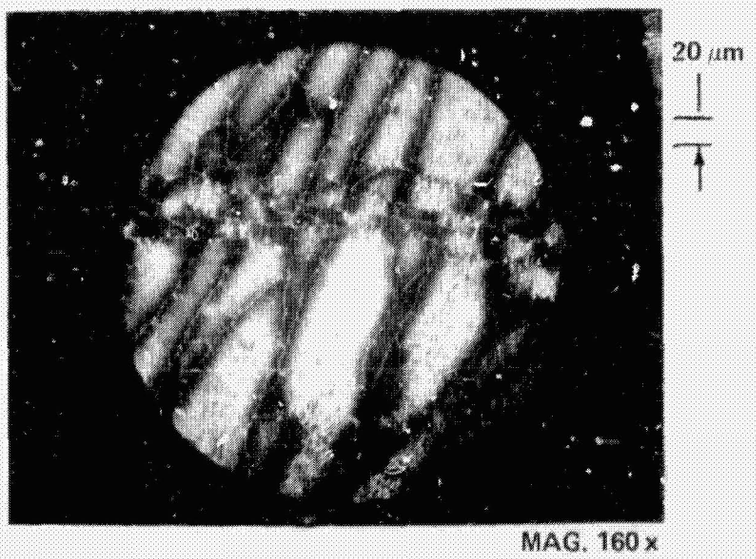


Figure 9. Photograph of Crazed and Fractured Portions of S/N 6. Magnified 160 x

S/N 12 blistered (as shown in Figure 7) very soon after initial pumpdown prior to thermal cycling, as expected. The blister increased in area, to perhaps 25% larger than shown, when heat was applied. Since this sample was bonded with Mystik #7366 that had not received prior aging before applying the Ag/teflon, it confirms qualitatively that prior outgassing or aging is essential to avoid bubbling. Specimens 3, 4, 5, 6, 7 and 8 were bonded with "outgassed" Mystik #7366 and experienced little or no bubbling or lifting.

S/N 9 and S/N 10, consisting of Ag/teflon plus 3M #467 adhesive, were visibly in the best condition after the test with no bubbling and only slight lifting evident during the test.

With the exception of S/N 1 discussed above, there was no delamination observable at the Ag/teflon interface for any sample. The control sample, S/N 5, which had previously failed the scotch tape test, survived the thermal cycling test. All of the commercially purchased material survived thermal cycling.

The effects caused by rapid compression, typified by Figure 9, are unexplained. The brighter "cloudy" areas of Figure 9 are caused by microscopically small fractured areas apparently coinciding with film portions that retained more rigid contact with the Al plate. This latter conclusion is drawn from the fact that the delibrately unbonded areas of S/N 11 did not show this microscopic fracturing. A further investigation of this phenomenon was considered outside the scope of this effort and is merely noted here for the sake of completeness. Although the unexpected venting produced the most evident defects to come out of the thermal test, they were not relevant to the test's defined objective.

F. Conclusions and Comments

For a quick summary of results and conclusions see Table 3.

1. Optical measurements performed on 21 selected specimens of Ag/teflon purchased from G. T. Schjeldahl Co. revealed that they possessed an average α equal to 0.085 with a standard deviation of ±0.005. Hemispherical emittance was found to be 0.76 (based on an average measured ϵ_n = 0.804). This gives an average α/ϵ = 0.112 with a standard deviation of ±0.006.

   From transmittance measurements performed on the test samples in the near uv, it is possible to conclude that evaporation conditions vary during the Ag and inconel depositions. For this reason α, ϵ and τ_λ should be measured on each sample prior to selection for spaceflight use.

Table 3

Summary of Test Results

Optical Measurements

- $\bar{\alpha}$ = 0.085 (with ±0.005 std. deviation among 21 samples).
- $\bar{\epsilon}$ = 0.76 (from ϵ_n = 0.804).
- $\bar{\alpha}/\epsilon$ = 0.112 (with ±0.006 std. deviation among 21 samples).

Scotch Tape Test

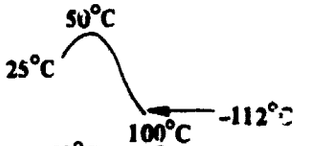
- All Schjeldahl supplied material passed.
- GSFC supplied control sample failed.

uv Degradation Test

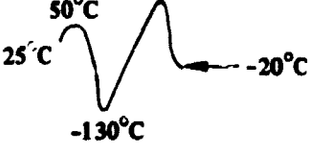
- All samples passed.
- Largest increase in α: +0.006 after 200 EUVSH.

Temperature Cycling Test (-130°C ≤ T ≤ +200°C)

- No delamination at Ag/teflon interface.
- Mystik #7366, 3M #467 } Passed
- Gelva 263 - Failed at:



- RTV 6-1104 - Failed at:



2. Both the "scotch tape" quick adhesion check and thermal cycling indicated that the commercial Ag/teflon possessed an adhesion at the Ag/teflon interface that was equal to or better than that of the material coated at GSFC.

3. Results of the uv degradation test indicate no significant difference in behavior between the control and test samples. Those samples supplied with 3M #467 adhesive already applied (specifically sample T1) showed a greater potential for degradation, although observed changes were well within acceptable limits.

   The samples were not exposed to a charged particle (e^-, p^+) environment as part of this test. An exposure such as this would be necessary to satisfy the needs of a variety of specific flight programs, but the uv irradiation test data was judged to be a sufficient means of revealing any potential degradation problems. A charged particle exposure would have strengthened but not altered the conclusions drawn from the present test.

4. The temperature cycling test confirmed the results of earlier work that demonstrated the acceptability of Mystik #7366 and 3M #467 adhesives as bonding agents for Ag/teflon over the temperature range from -130°C to at least +80°C and perhaps as high as +150°C. Because of thermally induced crazing and an undefined irradiation damage rate, it would be inadvisable to rely on Ag/teflon at temperatures exceeding 150°C. The results also indicated that some type of prior cure or outgassing is essential to eliminate bubbling and lifting in the adhesive. A 2 week cure in air was used in this test in accordance with past procedure but the interrelationship of time, temperature, pressure, humidity, etc. is essentially undefined and would benefit greatly from further work. A separate outgassing study would have to be performed to guarantee that these 2 adhesives can meet specific spacecraft acceptability criteria.

   The number of cycles experienced by the bonded samples was limited and long term aging effects remain undefined.

   Gelva 263 and RTV 6-1104, as formulated and applied in this test, were found to be unsuitable bonding agents for Ag/teflon.

5. The main conclusion that may be drawn from this series of tests is that the commercially purchased Ag/teflon behaves no differently than that which was formerly coated at GSFC. Therefore, all earlier test results, both flight and laboratory, and all practical experience acquired in working with the GSFC coated Ag/teflon can be applied to the Schjeldahl coating.

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APPENDIX

DISCUSSION OF SOLAR ABSORPTANCE MEASUREMENT

Because the solar absorptance of Ag/teflon is relatively low ($\alpha = 0.085$), even small measurement inaccuracies can produce a high percent error. For this reason, special attention was given to minimizing instrumental and procedural errors when determining α.

Spectral reflectance (ρ_λ) over the wavelength interval from 0.29μm to 2.4μm was measured using an integrating sphere reflectometer of the type described by Edwards, et al., and built by Gier-Dunkel Instruments.[18] The sample is located at the geometrical center of the integrating sphere normal to the equatorial plane; a 2.54 cm entrance aperture in the sphere wall on the equator faces the sample. A detector aperture containing a photomultiplier tube and a PbS cell is located at the sphere's south pole. Reflectance measurements were made with the sample rotated 20° off normal. The systematic errors inherent in this technique have been comprehensively treated in the literature and a conservative estimate of the total error is given as ±0.015.[18] If not further refined, this would result in a ±17.6% uncertainty in α for Ag/teflon. Experimental practice has established that α measurements can be repeated with a precision of ±0.003.

Further refinement in accuracy was achieved by comparing the reflectance of freshly evaporated Al measured on a high accuracy reflectometer of the Bennett-Koehler type[19] with data obtained from the same sample using the integrating sphere reflectometer. This procedure effected a calibration of the integrating sphere for specular samples that was applied to the specular Ag/teflon material in question.

To complete the reflectance measurements over the range of the solar spectrum beyond 2.4μm, a heated cavity (hohlraum) type reflectometer was used to measure total reflectance over the wavelength region from 2.4μm to 8.0μm. A description of this instrument and a discussion of its limitations can be found in Reference 20. Prior calibration using an evaporated Au sample of known reflectance had reduced the uncertainty in ρ_λ to ±0.02 in regions where ρ varies slowly with λ. Since there is only about 2% of the solar constant contained within the interval $2.4\mu m \leq \lambda \leq 8.0\mu m$, a measurement error of ±0.02 is insignificant, as will be demonstrated below.

The spectral reflectance of a 125μm (5 mil) Ag/teflon sample over the wavelength range $0.29\mu m \leq \lambda \leq 8.0\mu m$ obtained using the above instrumentation is given in Figure A.1. The measured specimen was from the control sample used in the qualification test discussed earlier. Approximately 99% of the total

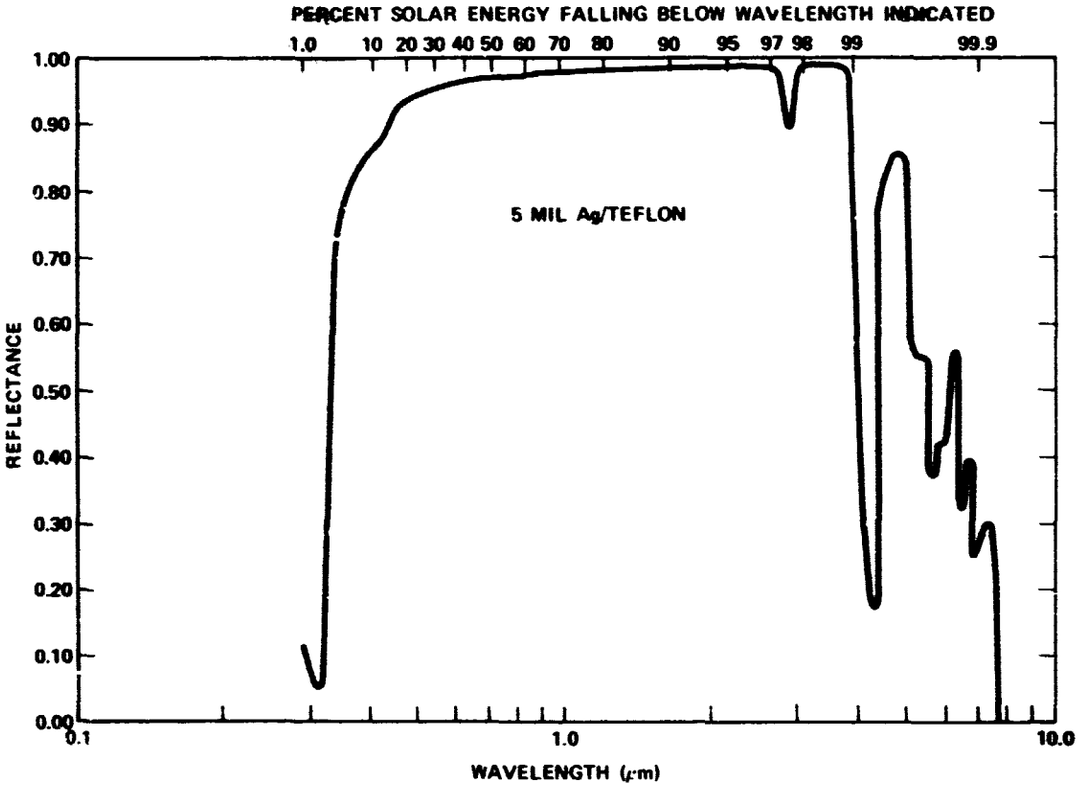

Figure A.1. Spectral Reflectance of 125μm (5 mil) Thick Ag/teflon over the Solar Wavelength Region ($0.29\mu m \leq \lambda \leq 8.0\mu m$)

solar spectral irradiance is contained within the measured bandwidth as indicated by the upper scale gradations in Figure A.1.

Solar absorptance was obtained from the spectral reflectance data of Figure A.1 by means of the familiar selected ordinate method of numerical integration. By definition:

$$\alpha = 1 - \rho = 1 - \int_0^\infty \rho_\lambda H_\lambda d\lambda \Big/ \int_0^\infty H_\lambda d\lambda$$

where H_λ is Johnson's solar spectral irradiance function,[21]

$$\int_0^\infty H_\lambda d\lambda = H$$

The integral

$$\rho = \int_0^\infty \rho_\lambda H_\lambda d\lambda / H$$

is in practice replaced by the summation:

$$\rho = \sum_{i=1}^{n} \rho_{\Delta\lambda_i} p_i$$

$$\text{where } p_i = H_{\Delta\lambda_i}/H \text{ and } \sum_i p_i = 1.$$

The procedural error in data reduction is minimized by making n large; a value of n = 100 was used for all α determinations in this study. Since n = 100, then $p_i = 0.01$ and each selected $\Delta\lambda_i$ encompasses 1% of the total solar energy.

The instrumental error, $\Delta\rho_\lambda$, appearing in each value of $\rho_{\Delta\lambda_i}$ must be similarly weighted to assess its ultimate impact on α, i.e.:

$$\Delta\rho_T = \left[\int_{\Delta\lambda_1} \Delta\rho_\lambda H_\lambda d\lambda + \int_{\Delta\lambda_2} \Delta\rho_\lambda H_\lambda d\lambda \right] \Big/ H$$

where $\Delta\lambda_1$ refers to the region measured by the integrating sphere reflectometer and $\Delta\lambda_2$ refers to the region measured by the heated cavity reflectometer.

Let $\Delta\rho_\lambda = \rho_\lambda - \rho'_\lambda$

where $\rho_\lambda \rightarrow$ integrating sphere reflectance data
$\rho'_\lambda \rightarrow$ high accuracy reflectometer data.

Experiment showed that $\Delta\rho_\lambda$ was never more than ± 0.003 for the evaporated Al standard and that:

$$\Delta\rho = \int_0^{2.4\mu m} \Delta\rho_\lambda H_\lambda d\lambda \Big/ H = -0.001.$$

For the region $\Delta\lambda_2 = 2.4\mu m \leqslant \lambda \leqslant 8.0\mu m\ (\infty)$, $\Delta\rho_\lambda \simeq 0.02$ for slowly varying ρ_λ. Since $H_{\Delta\lambda_2}/H \simeq 0.02$, then

$$\int_{\Delta\lambda_2} \Delta\rho_\lambda H_\lambda d\lambda \Big/ H \simeq 0.000 \text{ and was ignored.}$$

Therefore $\Delta\rho_T = -0.001$ and this essentially insignificant correction was applied to all Ag/teflon α data. For the control sample whose reflectance is plotted in Figure A.1:

$$\alpha = 1 - (\rho + \Delta\rho_T) = 1 - 0.916 + 0.001 = 0.085$$

As a conservative gesture, a tolerance of ± 0.010 was attached to the α data listed in Table 1.